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13. ABSTRACT (Maximum 200 words)

The aims of the project were to acquire metallic based amorphous metal alloy powder, consolidate the powder into bulk structural amorphous metal (SAM), blend the amorphous powder with crystalline Ta or W powders and consolidate the blends into bulk amorphous metal matrix particulate composite. Under proper consolidation conditions the SAM and SAM matrix composites were expected to exhibit shear localization under high strain rate conditions, the preferred failure mechanism in kinetic energy penetrator material. The results for amorphous Vitreloy 106a powder show that essentially full density can be achieved after one ECAE pass at temperatures of T_g and higher. Significant particle-to-particle bonding is evident from optical microscopy, Vickers microhardness indentations, mechanical testing and SEM analysis. Retention of amorphous character in the consolidate is demonstrated by XRD, DSC and shear banding. Effective consolidation is a result of the combined action of compaction and simple shear caused by the ECAE process. The results demonstrate that ECAE is a viable processing method for producing bulk amorphous metal (BAM) and BAM matrix – crystalline metal particulate composites.

14. SUBJECT TERMS	15. NUMBER OF PAGES
Amorphous metal, powder consolidation, kinetic energy penetrator, zirconium based amorphous metal, extrusion, equal channel angular extrusion (ECAE)	7
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TEES Final Report to ARO (DARPA) on the SAM Project: High Density Amorphous Metal Matrix Composites for Kinetic Energy Penetrators

May 5, 2005

by K.T. Hartwig and I. Karaman

1. Statement of the problem studied

Kinetic energy penetrators (KEP) are used for high-energy material experiments and to pierce armor. High-density materials such as tungsten (19.3 g/cm3) and depleted uranium (DU) (19 g/cm3) have been used for this application since the 1970's. DU is the material of choice today because of its very high-density, availability, pyrophoricity, superior ballistic performance, and low cost [1]. But since DU emits low-level radiation and is a heavy metal with toxicity hazards, it presents environmental concerns. The cost of clean-up and politics associated with DU urge R&D to improve the performance of tungsten alloys or create new materials to match or exceed the performance of DU. The present study proposed a new material system and fabrication methods for replacement of DU penetrators with materials that are environmentally benign and could offer a reasonably priced alternates with adequate mechanical properties.

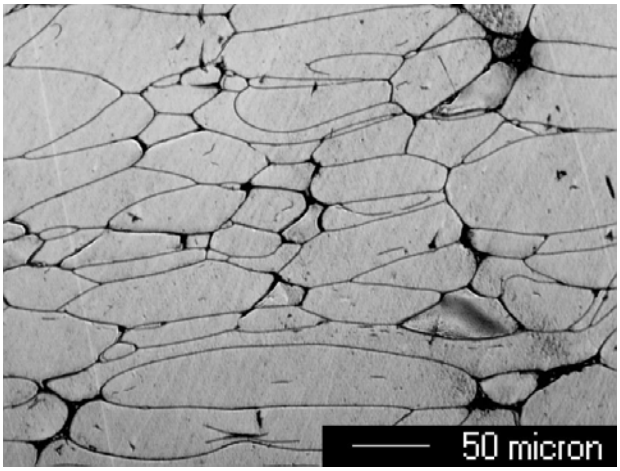
The material system chosen as the subject of this research would address four main properties desired for kinetic energy penetrators: high density, high strength, substantial ductility and self sharpening upon impact. Bulk metallic glasses (BMG) exhibit such properties and deform in a manner similar to DU, and therefore are promising candidate materials for DU replacement [2]. Most BMGs have been produced by rapid cooling from the melt (vacuum casting), but due to critical cooling rate requirements, material thickness is limited [3]. Two main powder consolidation techniques, conventional area reduction extrusion [4-7] and hot isostatic pressing (HIPing) [8-10], have been used to produce BMGs. HIPing is limited in its ability to achieve adequate particle-to-particle bonding and by long cycle times at high temperatures. Area reduction extrusion is limited by the resulting smaller cross section of the product.

Equal channel angular extrusion (ECAE) is a potential powder consolidation method that overcomes some of the difficulties associated with HIPing and conventional extrusion. The ECAE technique involves subjecting the powder to high pressure and simple shear in a die containing intersecting channels [11-13]. Potential benefits of the ECAE process are: constant workpiece cross-section, uniform deformation throughout workpiece and consolidation to near full density after a single extrusion [14]. The objective of the present study was to develop material systems and a processing method utilizing fabrication of amorphous alloy powders by high pressure gas atomization and consolidation of the powder by equal channel angular extrusion. The proposed investigations involved the development of a) a new high density amorphous alloy system, b) a new consolidation method for amorphous powders, c) a new glassy alloy plus dispersed crystalline phase composite and d) a better understanding of deformation and fracture mechanisms in bulk amorphous alloys. Improvements in material systems, and an understanding of the mechanical behavior of those systems for the penetrator application, will have a positive influence and impact on other potential applications of structural amorphous metals.

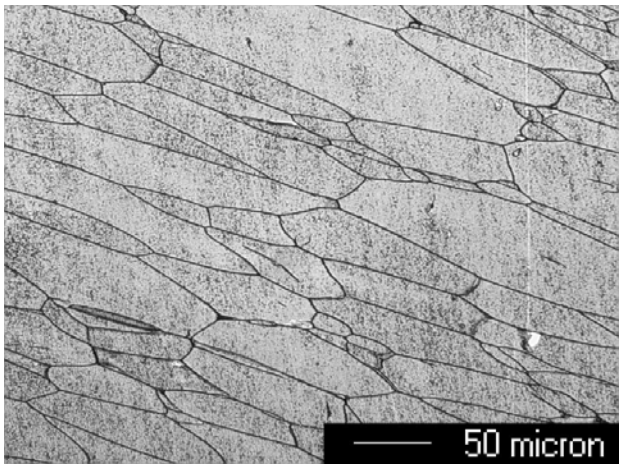
2. Summary of Important Results

The research done under this contract resulted in the development of material systems and a processing method utilizing fabrication of amorphous alloy powders by high pressure gas atomization and consolidation of the powder by equal channel angular extrusion. ECAE consolidated bulk amorphous metal and amorphous metal matrix composites resulting from this program show promise as materials for KEPs and other structural applications. Initial experiments were conducted on gas atomized Vitreloy 102 ($Cu_{50}Ti_{32}Zr_{12}Ni_5Si_1$) powder [15]. ECAE consolidation was conducted in the supercooled liquid region at a number of temperatures and extrusion rates. After ECAE processing the material has T_g and T_x values greater than those of the starting powder. The change in thermal properties is attributed to partial crystallization occurring from processing the material such that the TTT boundary is crossed. A high level of oxygen in the starting powder (1930 ppmw) likely causes nucleation sites for crystallization to occur. Early experiments showed ECAE to be a promising avenue for fabrication of BAM if the thermal processing characteristics of the amorphous powder could be improved and consolidation could be performed without crossing the time-temperature-transition boundary.

Because of the wider supercooled liquid region, lower glass transition temperature and the possibility of lower oxygen contamination, Vitreloy 106a ($Zr_{58.5}Nb_{2.8}Cu_{15.6}Ni_{12.8}Al_{10.3}$) was selected for further testing over Vitreloy 102 [16]. The gas-atomized Vitreloy 106a powder was produced from melting cast Vitreloy 106a ingots containing ~950 ppmw oxygen. After melting in a graphite crucible the powder was produced by high pressure gas atomization in argon. Inert gas fusion analysis showed the oxygen content of the powder (38 μm – 150 μm) to be approximately 1280 ppmw. T_g and T_x for the powder were 400°C and 460°C, respectively. This powder was consolidated using ECAE in the supercooled region at different strain rates and temperatures. The microstructure of all consolidates exhibited significant particle deformation as shown in Fig 1. The increase in aspect ratio of particles is seen to increase as the extrusion temperature increases. Some extrusions processed close to T_g show significant porosity. The consolidates exhibit a glass transition at approximately the same temperature as the powder. The exothermic peak on DSC curves shifts to slightly lower temperatures for all extrusions. This shift is attributed to the formation of a small fraction of nanocrystalline islands and to the thermal history effect. The decrease in T_x becomes more severe with increasing extrusion temperature. Extrusions at 440^{o}C show apparent crystallization supported by the lack of the exothermic peak on DSC traces as well as increased hardness and brittleness during Vickers microhardness testing. Shear banding is evident in hardness testing of the powder for all of the extrusions except the sample processed at 440 °C. There is an increase in the consolidate hardness depending on the extrusion temperature, however, there is not a direct correlation between the hardness values and the extrusion rate. Compression experiments demonstrate good consolidation and strength levels of 1500 – 1700 MPa comparable to those of cast V106 as shown in Fig 2. In spite of some nanocrystallization island and short range order formation upon processing, most of the fracture surfaces of the consolidates show shear banding and well-developed vein patterns, a typical fracture characteristics of metallic glasses with good ductility, as well as small amounts of interparticle debonding (See Fig 3). These results demonstrate that although high oxygen content in the initial powder restricts the allowable processing temperature



(a)



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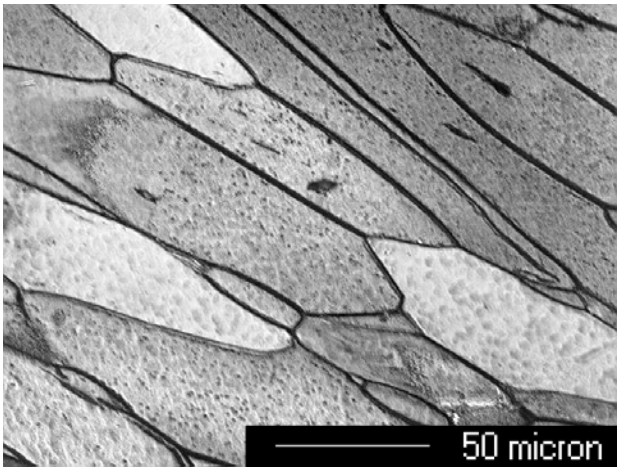

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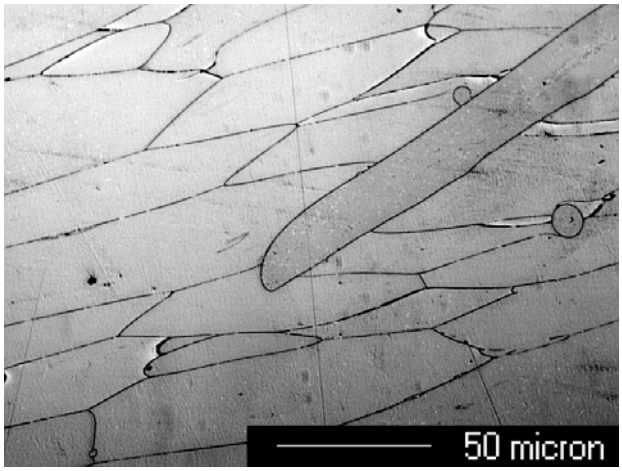


(d)

Fig 1: Optical micrograph of the flow plane of ECAE consolidated V106a with 1280 ppmw O_2, extruded at: (a) 400°C, (b) 420 °C, (c) 430 °C, and (d) 440 °C. The extrusion direction is in the flow plane which is the plane of the paper.

and rate, ECAE is successful in consolidation of metallic glass powder in the supercooled liquid region.

Modeling of the temperature rise during the ECAE processing was done by Tony Zarah of Matsys. The behavior of a solid copper bar was successfully done and compared with TEES experimental data. Three iterations of modeling were performed, with better results each time, but modeling of the Vitreloy 106a powder in a Ni can was never completed. It was concluded that insufficient funds were available for a comprehensive modeling effort.

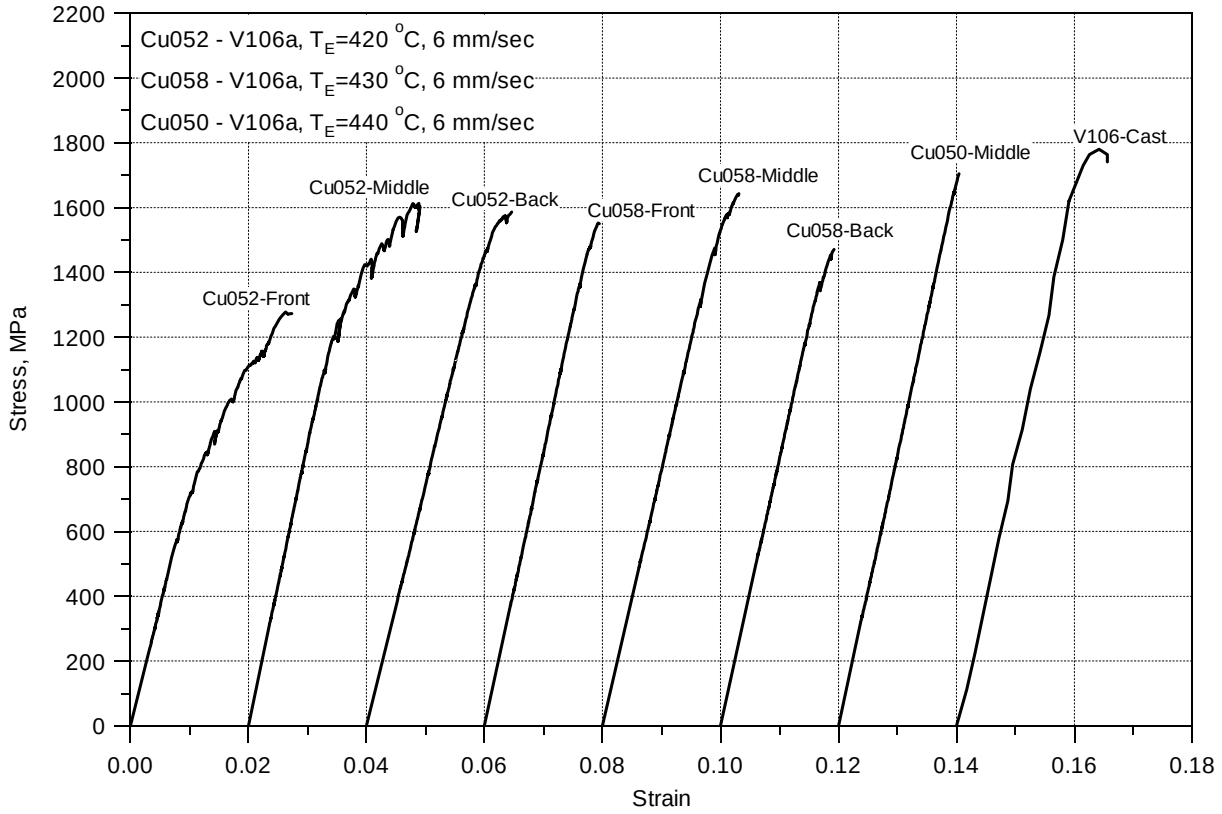
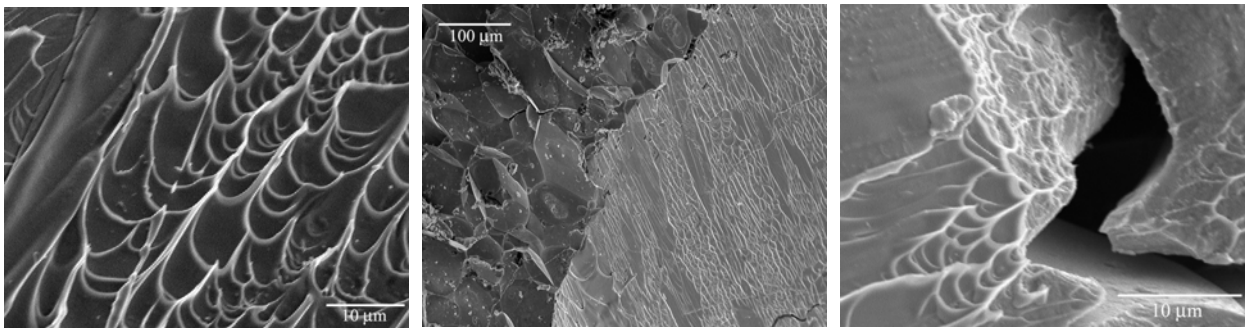


Fig 2. Compression experiments on the ECAE consolidated V106a samples and cast V106. The test specimens were 3 mm in diameter by 6 mm in length. The strain rate was 10^{-4}/s.



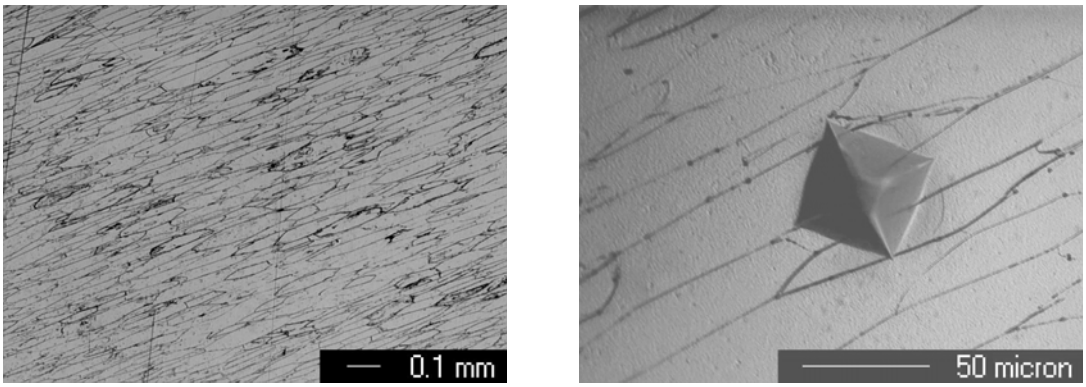
(a) (b) (c)

Fig 3. SEM of the fracture surfaces of V106a compression samples extruded at (a), (b) 430°C, and (c) 420 °C. Shear bands with well-developed vein patterns, shear slipped regions and interparticle debonding are apparent.

To remedy some of the problems caused by excessive oxygen contamination, a second batch of Vitreloy 106a powder was prepared from cast Vitreloy 106a buttons made by arc-melting pure elements, including low oxygen "crystal bar" Zr [17]. Inert gas fusion analysis showed the oxygen content of this new powder (38 μm – 150 μm) to be approximately 780 ppmw. Consolidation of this lower oxygen Vitreloy 106a (780 ppm) produced by better HPGA techniques verifies that essentially full density can be achieved after one ECAE pass at temperatures of T_g and higher. Significant particle-to-particle bonding is again evident from optical microscopy and Vickers microhardness indentations (See Fig 4). Thermal analysis by DSC shows the detrimental nature of oxygen to thermal processing of the materials at temperatures above T_g. The decreased oxygen contamination gives the benefits of a larger processing window, and a greater supercooled liquid region (ΔT). The added time and temperature from the lower oxygen content aid in consolidation by allowing a slower extrusion, which in turn causes less of an adiabatic temperature rise during processing. It also enables homogeneous material flow during extrusion and allows the carrying out of multiple extrusions. Retention of amorphous character is apparent for the low-oxygen consolidated powder both in shear banding surrounding microhardness indentations and by the lack of change between the DSC scans of the consolidated materials and the original powder. Fine cracks, suspected to be caused by cooling stresses, are present in the low oxygen Vitreloy 106a consolidate (See Fig. 4 a).

Because of the beneficial properties that the low-oxygen Vitreloy 106a powder demonstrated, it was selected to be blended with pure W to form amorphous metal matrix composite (AMMCs). Successful consolidation by multipass ECAE of the Vitreloy 106a with up to 70 vol.% W was accomplished in the supercooled liquid region. Microstructural observations show good infiltration of the Vitreloy phase in between W particles with some apparent W particle pull out (during polishing) (See Fig 6.). Retention of some ductility (i.e., no devitrification) in the Vitreloy matrix phase is demonstrated by shear banding in the Vitreloy phase around hardness indents. Substantial strength is demonstrated by the bonds between the W particles and Vitreloy phase as evidenced by no interfacial cracking from hardness indents and shear bands that cross prior particle boundaries. It was determined that the tungsten particles used were agglomerates, rather than individual particles like the amorphous phase precursor powder, which limited bonding in some regions, and resulted in pull-outs during metallographic polishing. While this affected the tensile properties (~400MPa tensile strength), compressive strengths are not affected significantly (~1.6 GPa compressive strength) and the fracture surfaces in the compression tests show evidence of glassy flow and shear localization (See Fig 7 and 8).

The results for amorphous Vitreloy 106a powder show that essentially full density can be achieved after one ECAE pass at temperatures of T_g and higher. Significant particle-to-particle bonding is evident from optical microscopy, Vickers microhardness indentations, mechanical testing and SEM analysis. Effective consolidation is a result of the combined action of compaction and simple shear caused by the ECAE process. The results demonstrate that ECAE is a viable processing method for producing BAMs and bulk glassy metal – crystalline metal composites.



(a) (b)

Fig 4. (a) Optical micrographs of flow plane for V106a (1080 ppmw O_2) extruded at 430°C and 0.5mm/s after one extrusion pass. The extrusion direction is left to right. (b)Vickers microhardness indentation for V106a (1080 ppmw O_2) extruded route 2B at 420°C and 0.5mm/s showing shear bands indicative of amorphous character. The sample verifies the predominant amorphous nature after two extrusions.

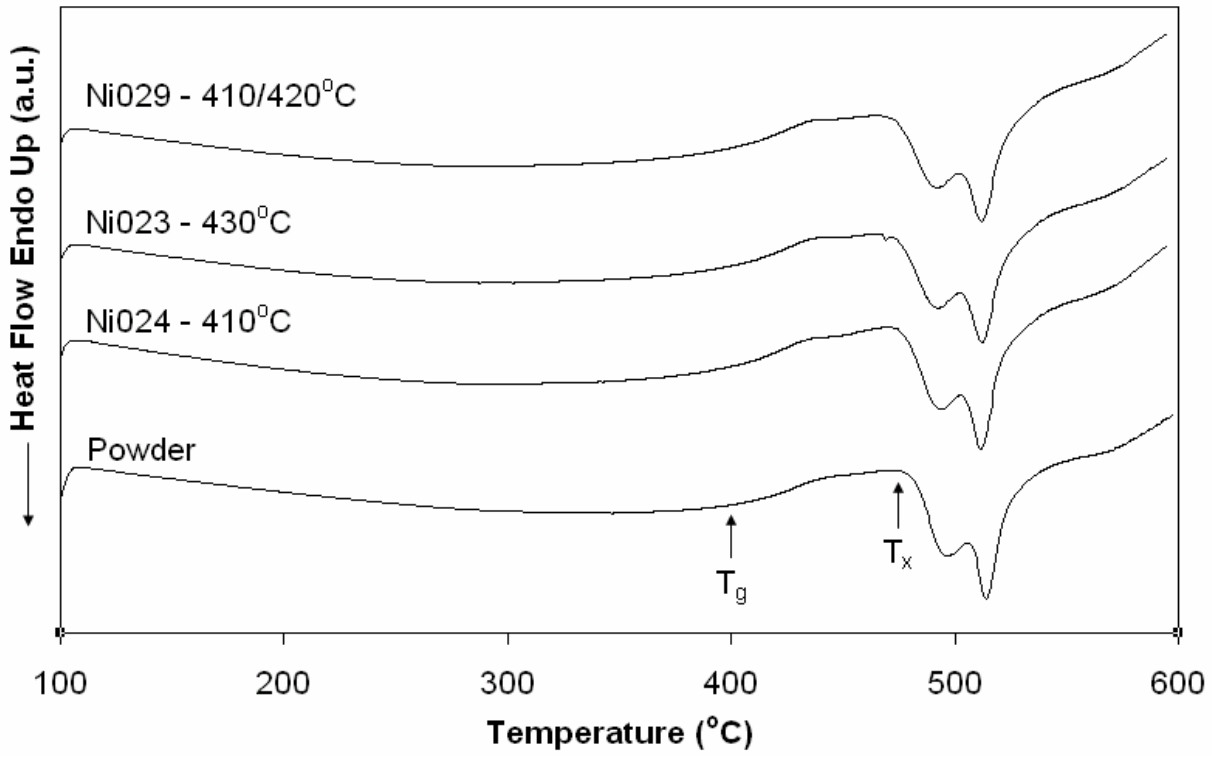
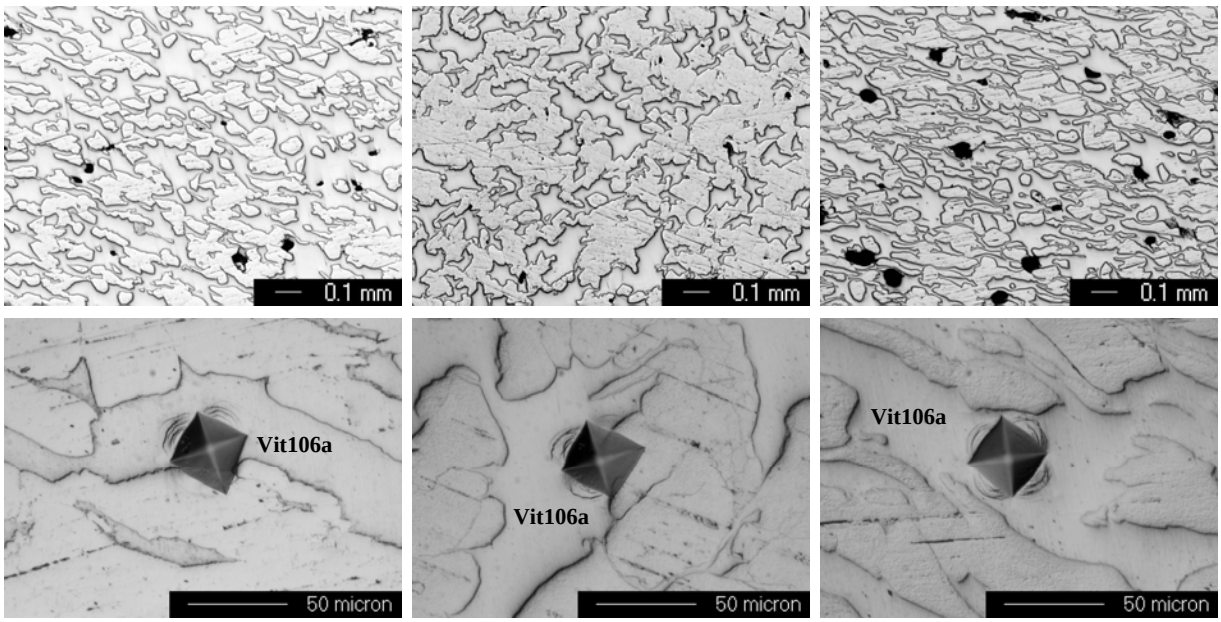


Fig 5. DSC scans of 1080 ppmw O_2 V106a consolidates. These show similar curves regardless of processing temperature indicating that the lower oxygen content increases thermal stability.



(a) (b) (c)

Fig 6. Flow plane optical micrographs of V106a and tungsten composites: (a) Route 1A, 50 vol% tungsten, (b) Route 2C, 60 vol% tungsten, and (c) Route 2B, 70 vol% tungsten

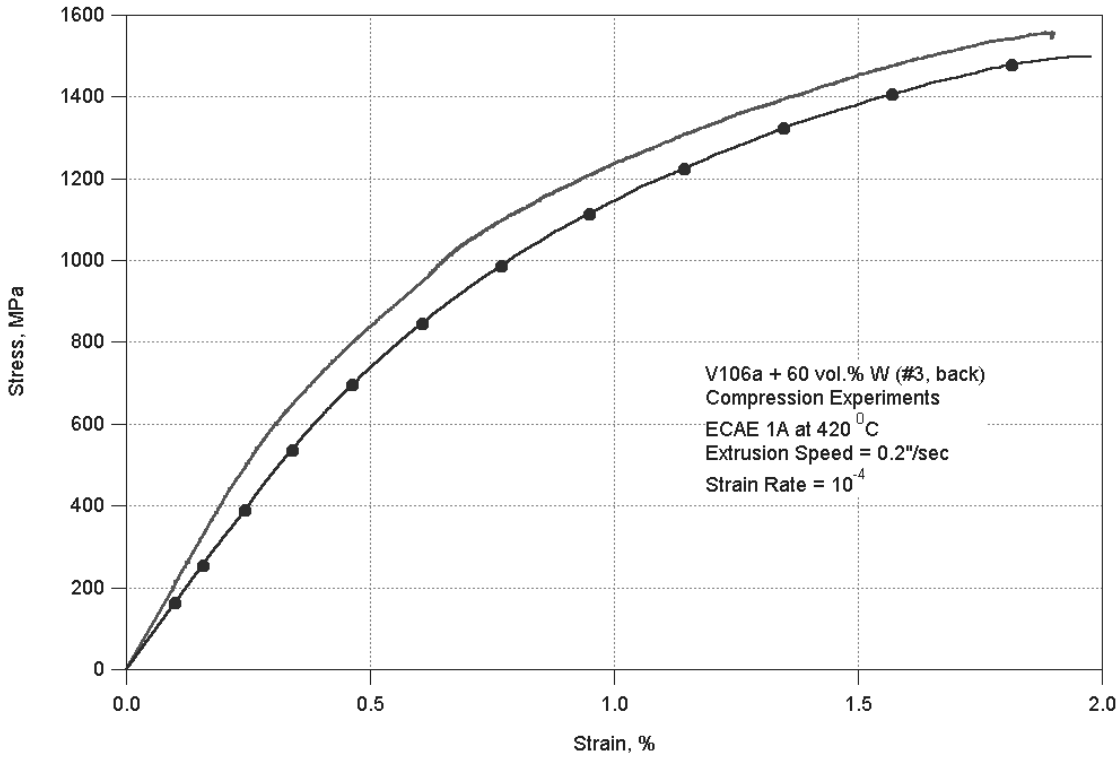


Fig 7. Compression test stress-strain curves for an ECAE consolidated V106a + 60 vol% W.

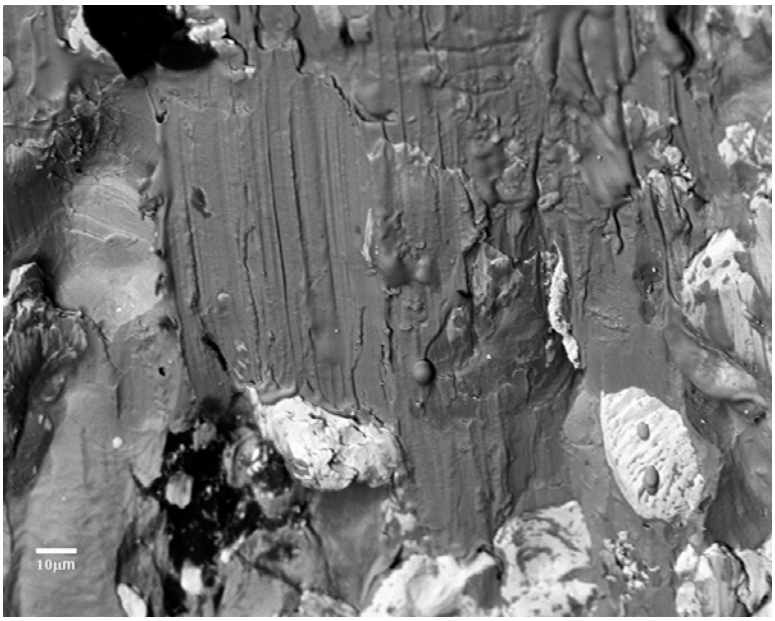


Fig 8. SEM of a fracture surface from the compression test of an ECAE consolidated V106a + 60 vol% W composite.

During the period of research a number of collaborative efforts were made with other DARPA/SAM project researchers. Bundles of cast Vitreloy 105 rods fabricated by Liquidmetal Technologies (LMT) were extruded via ECAE in attempts to consolidate the material. For this experiment 0.11 inch diameter by 1.9 inch long cylindrical rods were mechanically ground and chemically etched to remove the oxidized surface and packed into a Ni can. The as-received material had Tg and Tx values of 405°C and 465°C. Following single pass ECAE, the extrusion can was opened for post processing inspection and the deformed Vitreloy rods were easily separated indicating virtually no inter-rod bonding. The processed rods were fully compacted indicating substantial deformation without fracture. However, it is apparent that little movement occurred between the rods during the single pass extrusion, which would account for the absence of inter-rod bonding.

Another collaboration with LMT involved ECAE processing of several 0.25 - 0.31 inch diameter bars of W (70 vol. percent) infiltrated with Vitreloy 106a. There were four objectives for these experiments: first to see if ECAE processing could break apart the contiguous W matrix; second to see if separated rods or large fragments could be joined; third to see if the morphology of the microstructure could be altered, and fourth to see if the W phase could be work hardened. Microhardness testing of the as-received and single pass extruded material indicated some ductility (no cracking around the indentations) in the amorphous phase. In addition, the tungsten phase deformed substantially and bonded well to the Vitreloy. It is not clear whether or not the contiguous W matrix in the starting material was broken apart by the processing. Because the microhardness measurements showed an increase in hardness following ECAE processing, there is some chance that the tungsten phase was hardened. A disappointing

result of these experiments was that the split rods and chunks were not effectively joined by the ECAE processing. It is clear that processing conditions (temperature, strain and possibly hydrostatic pressure) were not adequate to cause significant adjoining surface disruption to encourage bonding of the large pieces.

Efforts to produce a porous, cell structured amorphous metal were made in collaboration with Dr. David Dunand (Northwestern University) [18]. Two ECAE runs were performed on blended (50 volume percent) Vitreloy 106a (1280 ppm oxygen) and NaCl powders. Results of evaluations by Dunand's group at Northwestern indicated that the salt did not interact with the Vit106a, the salt effectively flowed during extrusion and allowed the Vitreloy 106a particles to contact each other, deform and occasionally join, and the Vitreloy 106a remained fully amorphous during processing.

Some single-pass ECAE consolidation experiments were run with DAR19 iron-based amorphous metal powder supplied by Dan Branagan. The Tg reported for this material is 538°C; it does not exhibit a measurable Tg-Tx region, yet sufficient ductility is present below T_g for significant plastic deformation. A sample processed at 520°C showed some apparent consolidation in bands near the back of the billet. Close inspection of this banded region revealed that some consolidation had occurred, but only in regions with large (>10 μm) partially or fully crystalline particles embedded in an amorphous DAR19 matrix. Some porosity is evident as shown in Fig 9.

Additional consolidation experiments on iron-based amorphous metal powder were done on one of Joe Poon's alloys ($FeCr_{15}Mo_{12}Y_2C_{15}B_6$). This material is reported to have T_g amd T_x values of ~570^{o}C and ~595^{o}C. Atomized powder (30-150 μm) was vacuum encapsulated in Cu and Ni cans and extruded at 560, 580 and 590^{o}C at a punch speed of 25 mm/s. the material encapsulated in Ni and processed at 580^{o}C was partially consolidated as shown in Fig. 10. DSC analysis indicates partial devitrification of the 580^{o}C consolidate, but microhardness indents

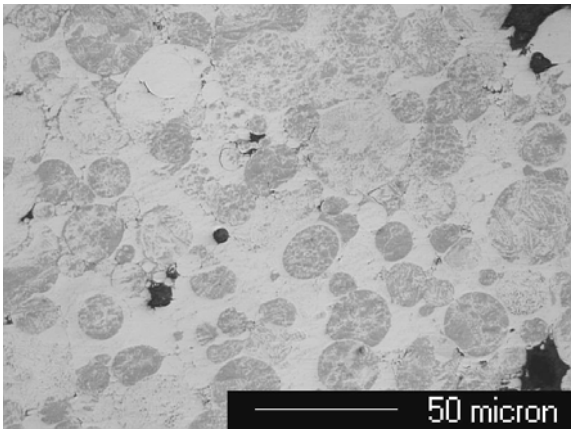


Fig 9. Optical micrograph of ECAE consolidated DAR 19 showing residual porosity.

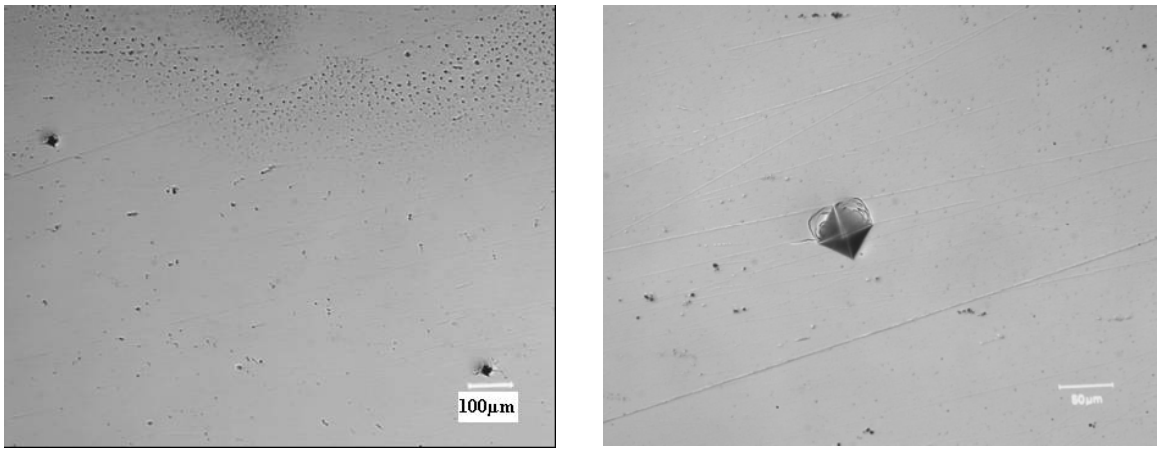


Fig 10. Flow plane of sample extruded in Ni can at 580°C and 1"/sec. The Vickers microhardness indentations (500g/13s) show shear banding demonstrating the retention of amorphous character after consolidation. Average Vickers hardness of the consolidate was 1228±37. Hardness of the Ni can was 328±34.

show shear banding. Examination of the consolidated regions including areas of substantial porosity show evidence of good particle-to-particle bonding by virtue of a "wetted" network appearance. This judgment is reinforced by polished/etched samples on which no prior particle boundaries are visible.

A summary of the results on consolidation of amorphous metal powder is presented in Table 1.

Table 1. Summary of material characterization measurements on consolidated amorphous metal particulate.

Amorphous Metal Particulate	Tg/Tx (°C)	Crystalline Powder Blend	T of ECAE (°C)	Apparent Density (%)	Consolidated Hardness (HV500)	X-Ray Analysis	DSC Trace
V102	427/480	---	430-450	95-99	540-615	Amorphous	Small shift
V105	403/464	---	400	99	512	---	---
V106a	400/480	---	400-430	98-99	470-510	Amorphous	No shift
V106a	400/480	W	400-430	98-99	490-515	Amorphous	---
DAR19	583	---	490	80-99	---	---	---
Poon Alloy	570/595	---	560-590	80-99	1230	---	Shift

This DARPA project is judged partially successful by the investigators. The positive contributions include:

a) Realization of the importance of powder with low oxygen contamination and a clean surface region to enhance particle-to-particle bonding and inhibit crystallization.
b) Development of successful ECAE processing equipment/strategies that include isothermal tooling, nonuniform billet heating, adiabatic heating during deformation and TTT boundary considerations.
c) Realization of the importance of amorphous alloys with a substantial subcooled liquid (T_x-T_g) region combined with a low T_g. Alloys with a low T_g tend to also be more stable against devitrification, and thus offer a longer processing window, because of slowed reaction kinetics.
d) Demonstration of consolidation to near full density of amorphous metal powder by ECAE at temperatures near T_g to give bulk structural amorphous metal.
e) Determination of key processing variables for effective ECAE consolidation which include temperature (near T_g), strain rate (<10 s^{-1} and dependent on T), pressure (dependent on T) and time-temperature history (~90% of time to TTT boundary).
f) Determination that infiltration between crystalline particles by amorphous metal phase is effectively accomplished via ECAE.

The work remaining that is necessary to demonstrate the effectiveness of a dense metal matrix composite fabricated by ECAE for kinetic energy peneratrator applications includes:

a) Demonstration of excellent metallurgical bonds between amorphous particles and between the amorphous and dense crystalline (W) phase.
b) Elimination of macro scale cracking in the consolidate. Such cracking is thought be consequence of either non uniform stress and plastic strain during ECAE processing or non symmetric thermal stresses present during extrudate cool down (from processing temperature).
c) Fabrication of samples of sufficient size for ballistic testing.
d) Demonstration of self-sharpening behavior during ballistic impact with armor.

The investigators believe that the work remaining can be accomplished within a reasonable period of time, and that ECAE consolidation of amorphous metal plus crystalline powder blends into very large penetrators (25mm diameter by 400mm long) is possible.

3. Listing of all publications and technical reports supported under this grant or contract

(a) Papers published in peer reviewed journals

1. J. Robertson, J.-T. Im, I. Karaman, K.T. Hartwig and I.E. Anderson, "Consolidation of amorphous copper based powder by equal channel angular extrusion**",** Journal of Non-Crystalline Solids, Volume 317, Issues 1-2, March 2003, Pages 144-151
2. I. Karaman, J. Robertson, J-T. Im, S.N. Mathaudhu, Z.P. Luo and K.T. Hartwig, "The effects of temperature and extrusion speed on the consolidation of zirconium-based metallic glass powder using equal-channel angular extrusion", Metallurgical and Materials Transactions, A., Volume 35A, pp. 247-256. 2004.

(b) *Papers published in non-peer-reviewed journals or in conference proceedings*

1) S.N. Mathaudhu, J.-T. Im, R.E. Barber, I.E. Anderson, I. Karaman, and K.T. Hartwig, "Progress in consolidation of amorphous Zr-based powder into bulk metallic glass",

in Proceedings of the 2002 MRS Fall Meeting Symposium on Supercooled Liquids, Glass Transition, and Bulk Metallic Glasses, edited by A.L. Greer, T. Egami, A. Inoue, S. Ranganathan, MRS Publications, Vol. 754, pp. 191-198, 2003.

2) K.T. Hartwig, I. Karaman, M. Haouaoui and S.N. Mathaudhu, "Consolidation of Cu and amorphous Zr-based powders by severe plastic deformation", in Proceedings of the 2003 NATO Advanced Research Workshop: Metallic Materials with High Structural Efficiency, Kiev, Ukraine, September 6-13, 2003.

(c) *Papers presented at meetings, but not published in conference proceedings*

1) K.T. Hartwig, I. Karaman, S.N. Mathaudhu and J.-T. Im, "Consolidation of powders by severe plastic deformation", in Processing, Microstructure and Properties of Powder-Based Materials, TMS Annual Meeting, Charlotte, NC, March 14-18, 2004.
2) K.T. Hartwig, I. Karaman and S.N. Mathaudhu, "Consolidation of blended powders by severe plastic deformation to form amorphous metal matrix composites", in Bulk Metallic Glasses, TMS Annual Meeting, Charlotte, NC, March 14-18, 2004.
3) K.T. Hartwig, I. Karaman, S.N. Mathaudhu and M. Haouaoui, "Consolidation of advanced powders by severe plastic deformation", in Metallic Materials with High Structural Efficiency, NATO Advanced Research Workshop, Kiev, Ukraine, September 6-13, 2003.
4) K.T. Hartwig, I. Karaman, J.N. Robertson, S.N. Mathaudhu, J.-T. Im and I.E. Anderson, "Consolidation of zirconium based metallic glass powder by equal channel angular extrusion", in International Symposium on Intermetallic and Advanced Metallic Materials - A Symposium Dedicated to Dr. C. T. Liu, TMS Annual Meeting, San Diego, CA, March 2-6, 2003.
5) S.N. Mathaudhu, J. -T. Im, J.N. Robertson, R.E. Barber, I. Karaman, I.E. Anderson and K.T. Hartwig, "Consolidation of a glassy metal and crystalline powder blend by ECAE", in International Symposium on Intermetallic and Advanced Metallic Materials - A Symposium Dedicated to Dr. C. T. Liu, TMS Annual Meeting, San Diego, CA, March 2-6, 2003.
6) K.T. Hartwig, I. Karaman, J.N. Robertson, R.E. Barber, S.N. Mathaudhu, J.-T. Im and I.E. Anderson, "Consolidation of zirconium-based amorphous powder into bulk glass", in Supercooled Liquids, Glass Transition, and Bulk Metallic Glasses, MRS Fall Meeting, Boston, MA, December 2-6, 2002.
7) K.T. Hartwig and S.N. Mathaudhu, "Consolidation of Metallic Powders by Equal Channel Angular Extrusion", in the Powder Materials: Current Research & Industrial Practices, TMS Fall Meeting, Indianapolis, IN, November 4-8, 2001.
8) I. Karaman, K.T. Hartwig, S.N. Mathaudhu and I.E. Anderson, "Consolidation of Zr-based amorphous metal powders with equal channel angular extrusion", in Full Density Powder Processing, TMS Fall Meeting, Indianapolis, IN, November 4-8, 2001.

(d) *Manuscripts submitted but not published*

Additional papers are expected.

(e) *Technical reports submitted to ARO*

Many monthly reports.

4. List of all Participating Scientific Personnel showing any Advanced Degrees Earned by them while employed on the Project

a) Dr. K. Ted Hartwig (PI): Texas A&M University – Consolidation processing
b) Dr. Ibrahim Karaman (Co-PI): Texas A&M University – Material characterization and mechanical testing
c) Dr. Iver Anderson: Ames Laboratory- Material design and fabrication
d) Robert Barber (Research Associate/Engineer): Texas A&M University – Tool design and consolidation processing
e) Suveen N. Mathaudhu (Research Assistant) Texas A&M University – consolidation processing and material characterization
f) Jae-Taek Im (Research Assistant) Texas A&M University – Tool and material characterization
g) Jonathan Robertson (Research Assistant) Texas A&M University – Materials characterization: Earned M.S. under project
h) Tony Zarah: Matsys – Deformation modeling

5. Report of Inventions

None

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7. Appendices

None